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New validated RP-HPLC method for the determination of fexofenadine in bulk and dosage form

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ABSTRACT

This investigation describes a new precise, sensitive and accurate RP-HPLC method for the estimation of Fexofenadine in Bulk and Tablets. The resolution of drug was achieved on Symmetry C_{18} (150mm x 4.6mm i.d., 5µm particle size) column with UV detection at 254 nm and the mobile phase consist of Buffer and Methanol (30:70v/v).Using chromatographic conditions described Fexofenadine was well resolved with mean retention time of 3.399 min, respectively. Linear response (r>0.999) was observed over the range of 20-60µg/mL. The lower limit of quantification and lower limit of detection was found to be 9.92 and 3.03 for Fexofenadine. The Validation parameters were performed according to the ICH guidelines and the proposed method can be useful in the routine analysis for the determination of Fexofenadine in Pharmaceutical dosage forms.

Key Words: Fexofenadine, RP-HPLC, Symmetry column, Validation parameters, ALLEGRA.

INTRODUCTION

Fexofenadine (FEXO) is a histamine H1-receptor antagonist. The chemical name is (±)-4-[1 nylmethyl)-1-piperidinyl]-butyl]-α, α-dimethylhydroxy-4-benzene acetic acid. Used in the treatment of hay fever and similar allergy symptoms. Literature survey reveals many methods for FEXO individually and with the other drug combinations by Spectrophotometry, HPLC and Ion pair complexation. In this communication, a new simple, rapid and precise HPLC method has been reported for determination of FEXO which can be used for its routine analysis in normal laboratories.

MATERIALS AND METHODS

Chromatogram was made on Waters (Alliance) with Auto Sampler and Ultraviolet detector. The data acquisition was performed by Empower Software. Glass wares used in each step were rinsed thoroughly with double distilled water, dried in hot air oven. FEXO were obtained from Alekya drugs Ltd., Vijayawada. The pharmaceutical preparation of FEXO was ALLEGRA (Aventis Pharma Ltd.India.). Methanol used is HPLC grade obtained from MERCK (India) and water used is double distilled water. Other reagents were of AR grade.

CHROMATOGRAPHIC CONDITIONS

The used analytical column was symmetry C_{18} (150mm x 4.6mm i.d., 5µm particle size) column. The mobile phase consists of mixture of Buffer and Methanol (30:70 v/v), filtered through 0.22µm Millipore filter and degassed by sonication. Separation was carried out isocratically, at ambient temperature (23±1°C), and a flow rate of 1.0mL/min with Ultraviolet detection at 254 nm. The injection volume was 20 µL.

PREPARATION OF STANDARD SOLUTION

Accurately weigh and transfer 10 mg of FEXO into a 10mL clean dry volumetric flask separately. Add about 7mL of Diluent (Mobile phase) and sonicate to dissolve completely and make volume up to the mark with the same solvent (Stock solution).Further pipette 0.4mL FEXO from the above stock solution into a 10mL volumetric flask and dilute up to the mark with diluent.

ANALYSIS OF MARKETED FORMULATION

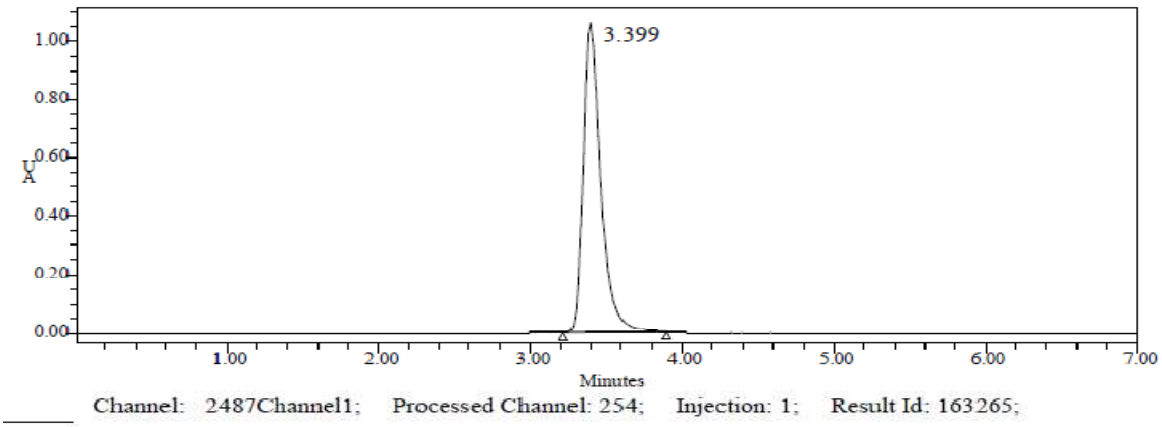
Twenty tablets of FEXO were crushed and made into powder. Accurately weigh and transfer equivalent to 10mg of sample into a 10mL clean dry volumetric flask. Add about 7mL of Diluent and sonicate to dissolve it completely and make volume up to the mark with the same solvent (Stock solution) and mix well and filter through 0.45µm filter. From the above stock solution, 0.4mL of FEXO was transferred into a 10mL volumetric flask and dilute up to the mark with diluent.20 µL of the standard, sample were injected into the chromatographic system and the areas were measured for the FEXO peaks. The content of FEXO was calculated and found to be 99.4% respectively.

RESULTS

Optimization of Chromatographic Conditions

Chromatographic conditions were optimized by changing the mobile phase composition and buffers used in the mobile phase. Different experiments were performed to optimize the mobile phase, but adequate separation of drugs could not be achieved. By altering the pH of mobile phase a good separation was achieved. The optimized mobile phase consisting of 7g of Potassium dihydrogen Phosphate in 1000mL of double distilled water (pH 3.0 with Orthophosporic acid) and Methanol mixed in the ratio of 30:70v/v and flow rate of 1.0mL/min, FEXO were eluted at 3.399min with a run time of 7min, under the above optimized chromatographic conditions. Typical chromatograms for estimation of FEXO had shown in Figure 1 and 2.

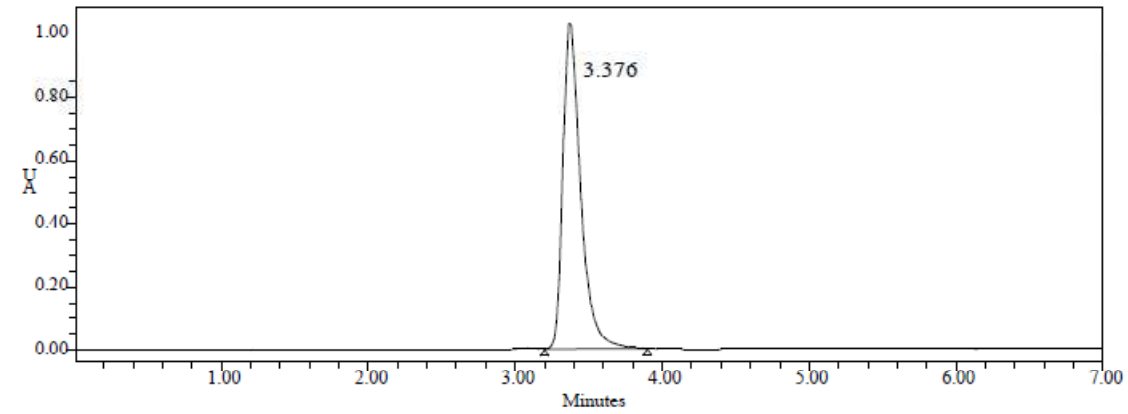
Figure 1: Chromatogram of FEXO Standard



Peak Name:Fexofenadine

	Peak Name	RT	Area	Height (µV)	USP Plate Count	USP Tailing
1	Fexofenadine	3.399	8705913	1058515.9	4132.5	1.4

Figure 2: Chromatogram of FEXO Sample

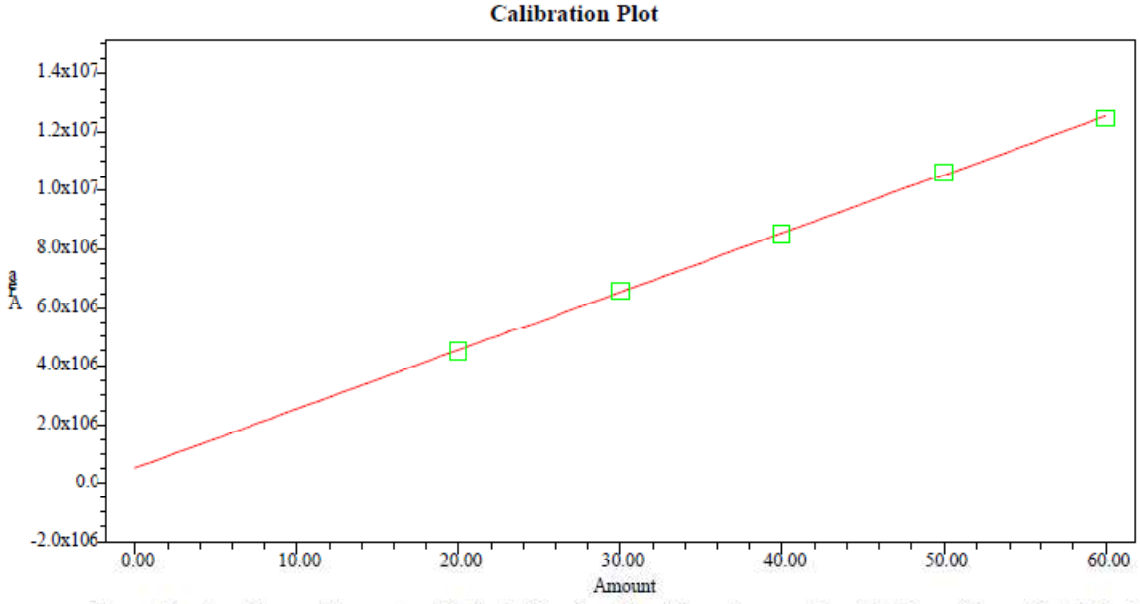


Channel: 2487Channel1; Processed Channel: 254; Injection: 1; Result Id: 163305; Processing Method. FexofenadineAssay spl

Peak Name: Fexofenadine

	Peak Name	RT	Area	Height (µV)
1	Fexofenadine	3.376	8599042	1038143.9

Figure 3: Calibration plot of FEXO



Name: fexofenadine; Processing Method: Fexofenadine Linearity curve2; Fit Type: Linear (1st Order);R^2: 1.000; R1.000

METHOD VALIDATION

System Suitability Results

For FEXO peak, the tailing factor was found to be 1.4 and the Theoretical Plates obtained were found to be 4132.5 respectively.

Linearity

The calibration curve was obtained by plotting Peak Area against Concentration for FEXO. The linearity was obtained in the concentration range of 20-60µg/mL for FEXO. The regression coefficient values (R^2) for FEXO was found to be 0.999 respectively. The linearity curve were shown in Figure.3

Accuracy and precision

The accuracy of the RP-HPLC method was determined by calculating Recovery of FEXO for 50%, 100% and 150% with respect to target concentration and results are tabulated in Table 1. The System precision of the proposed method was determined by injecting standard solution for five times and measured the area for them in HPLC. The Method Precision of the proposed method was determined by injecting six sample solutions into HPLC prepared individually. The %RSD for the areas of system precision and method precision data were calculated and given in Table 2.

Table 1: Recovery Results for FEXO

%Concentration (at specification Level)	Area	Amount Added (*mg)	Amount Found (mg)	**% Recovery	Mean Recovery
50%	4408918	5.0	5.06	101.3%	99.6%
100%	8555394	10.0	9.83	98.3%	
150%	12956839	15.0	14.8	99.3%	

***Average of three determinations (n=3)*

**Milligram*

Table 2: Precision of FEXO

S.NO.	PRECISION	FEXO
1.	System precision (Average Area and %R.S.D)	8341899 and 0.65
2.	method precision (Average Area and %R.S.D)	8596808and 0.30

Limits of Detection and Quantitation

For determining the limit of detection (LOD), 10mg of FEXO was transferred in 10mL clean dry volumetric flask separately. Add about 7mL of Diluent and sonicate to dissolve it completely and make volume up to the mark with the same solvent (Stock solution).

From this a working standard of 0.05µg/mL was prepared and injected. The LOD was found to be 3.03 for FEXO. For determining the limit of Quantitation (LOQ), from the above stock solution, prepared 0.01µg/mL solution of FEXO and injected. The LOQ was found to be 9.92.

Robustness

Robustness, a deliberate change in the flow rate, mobile phase composition was made to evaluate the impact on the method. The results reveal that the method is robust enough. The results are summarized in Table 3&4.

Table 3: System Suitability results for FEXO (Flow Rate Varied)

S.No	Flow Rate (ml/min)	System Suitability Results	
		USP Plate Count	USP Tailing
1	0.8	4155.0	1.5
2	1.0	4069.4	1.5
3	1.2	3793.8	1.5

Table 4: System Suitability results for FEXO (Mobile Phase Varied)

No	Change in Organic Composition in the Mobile Phase	System Suitability Results	
		USP Plate Count	USP Tailing
	10% less	4317.2	1.5
	*Actual	4069.4	1.5
	% more	3656.9	1.4

***** *Results for actual Mobile phase composition (Buffer and Methano (30:70 v/v)) have been considered from Accuracy standard*

CONCLUSION

A new HPLC method was developed and validated for determination of FEXO in pharmaceutical dosage form and assured the satisfactory precision and accuracy and also determining lower concentration of each drug in its solid dosage form. The method has been found to be better as no methods are reported, because of use of a less economical and readily available mobile phase, lack of extraction procedures, no internal standard and use of the same mobile phase for washing of the column. All these factors make this method suitable for quantification of FEXO in bulk drugs and in pharmaceutical dosage forms. It can therefore be concluded that use of the method can save much time and money and it can be used in small laboratories with very high accuracy and a wide linear range.

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REFERENCES

[1] British pharmacopoeia 1& 2, **2009**, 1-6.
[2] S Amin, S. Ahmed and A. Mohamed, *Journal of Chemical Engineering & Process Technology*, 1, 1-4.
[3] L.D Srinivas, P.Ravi Kumar and B.S.Sastry, *E-Journal of Chemistry*, **2005**, 2, 8, 199-202.
[4] Mohie Khaled Sharaf El-Din, Fawzia Ahmed Ibrahim et al. *Acta Chim. Slov*. **2011**, 58, 278–287
[5] PV Polawar, UD Shivhare, KP Bhusari and VB Mathur, *Research J. Pharm. and Tech*., **2008**, 1,4,539-541
[6] R Vijay Amrithraj,s. Purna Chander , 0.Jabir Aboobacker and Anudeep M,**2011**,2,2,738.
[7] MS Arayne, N.Sultana, AZ,Mirza, FA.Siddiqui, *J Chromatogr Sci*.,**2010**,48,5,382-5.
[8] K Suresh kumar,V.Ravichandran,M.K.Mohan muruga raja,R.Thygu, *Indian journal of pharmaceutical sciences*, **2006**,68,6,841-842.
[9] SM Pathak, AR.Kumar, P.Musmade, N.Udupa, Epub, **2008**, 76, 2,338-46.
[10] I Kozan, Palabiyik, E.karacan, F.onur, *Turk J. Pharm. Sci*., **2008**, 5, 3,175-189.
[11] J Deepak kumar,A Maulik kumar,J Deepti, *Indian J.Pharm,Educ.Res*.,**2010**,44,3,295-300.
[12] S Abd el-hay, Y el-mammli,A shalaby,*RJPBCS*,**2011**,2,1,497.
[13] A R Breier,M Steppe,E E S Scjapoval, *Analytical letters*,**2007**,40,12,2329-2337.
[14] U Hofmann,M Seiler,S Drescher,MF Fromm, *J Chromatogr B Analyst Technol Biomed Life Sci.*, **2002**,25,766,2,227-33.
[15] T Radhakrishna,*J Pharm Biomed Anal*,**2002**,29,681-690.
[16] RV Nirogi,VN Kandikere,M Mudigonda, K Komameni,*Rapid Commun Mass Spectum*,**2006**,20,3030-8.
[17] A R Brier,C S Stepe M Schapoval,*J Pharm pharmaceut.Sci*.,**2005**,8,2,289-298.
[18] P Mikus,I Valaskova, E Havranek,*Drug Dev.Ind.Pharm.*,**2005**,31,8,795-801.
[19] S S Zarapkar,N P Bhandari,U P Halkar,*Indian Drugs*,**2000**,37,9,421-425.
[20] I Fu,E J Woolf,B K Matuszewski, *J Pharm.Biomed.Anal.*,**2004**,35,837-846.